

**Phase separation in oxygen doped  $\text{La}_{2-x}\text{Sr}_x\text{NiO}_{4+\delta}$  ( $0.02 \leq x \leq 0.12$ ).**

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Beamline: X7A

**Introduction:** Oxygen intercalation in  $\text{La}_2\text{NiO}_{4+\delta}$  causes a number of interesting effects, as for example phase separation and ordering of the interstitial oxygen ions. Starting from stoichiometric  $\text{La}_2\text{NiO}_4$ , with increasing  $\delta$  first disordered oxygen phases and then oxygen ordered phases with different staging types of the interstitial oxygen layers were observed. In the disordered oxygen regime for certain values of  $\delta$  the system phase separates into oxygen rich and oxygen poor phases. Correspondingly one can find pure and mixed structural phases of the LTO (*Abma*) and the LTT (*P4<sub>2</sub>/ncm*) type. Excess oxygen also causes a doping of the  $\text{NiO}_2$  planes with charge carriers. For particular concentrations the charge carriers form a static stripe pattern with an incommensurability that is independent of the interstitial oxygen superstructure and similar to the charge stripe order in Sr doped  $\text{La}_{2-x}\text{Sr}_x\text{NiO}_4$ .

Recently, in  $\text{La}_{2-x}\text{Sr}_x\text{NiO}_4$  with  $x=0.04$  a similar biphasic LTO+LTT phase as in  $\text{La}_2\text{NiO}_{4+\delta}$  with  $\delta \sim 0.02$  was found. As the charge carrier content  $p=x+2\delta$  in these two samples is supposed to be the same, the observation raises the question whether the phase separation is driven by the charge carriers, the excess oxygen, or both.

**Experiment and Results:** Series of  $\text{La}_{2-x}\text{Sr}_x\text{NiO}_{4+\delta}$  samples with various amount of excess oxygen  $\delta$  have been prepared for fixed Sr content  $x=0.02, 0.04, 0.08$ , and  $0.12$ . To adjust  $\delta$  small pieces were annealed at 1000K in atmospheres with different partial oxygen pressure, the fugacity  $f_{\text{O}_2}$  ranging from -12 to 0. Synchrotron x-ray powder diffraction patterns were collected at beamline X7A at a wavelength of  $\lambda=0.7\text{\AA}$ . Temperature was controlled using a closed-cycle He displacer refrigerator. Powder samples were contained in glass capillaries ( $\phi 0.4\text{mm}$ ) sealed under argon. Rietveld refinements were carried out using Rietica. So far, mainly samples with Sr content  $x=0.04$  have been studied. Fig. 1 shows the temperature dependence of the lattice parameters  $a, b, c$  for biphasic  $\text{La}_{1.96}\text{Sr}_{0.04}\text{NiO}_{4+\delta}$ . At low T no clear evidence for a transition LTO  $\rightarrow$  LTLO (*Pccn*), as observed in biphasic  $\text{La}_2\text{NiO}_{4+\delta}$ , was found in our sample [1]. In Fig. 2 we plot the lattice parameters of  $\text{La}_{1.96}\text{Sr}_{0.04}\text{NiO}_{4+\delta}$  at room temperature as a function of  $f_{\text{O}_2}$ . With increasing  $f_{\text{O}_2}$  (increasing  $\delta$ ) we find the same sequence LTO  $\rightarrow$  LTO+LTT  $\rightarrow$  LTT as in  $\text{La}_2\text{NiO}_{4+\delta}$  (the chemical analysis of  $\delta$  is in process).

**Future plans:** To work out a conclusive phase diagram of  $(x, \delta, T)$  further samples have to be studied at X7A.

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**References:** [1] J. M. Tranquada et al., Phys. Rev. B 50, 6340 (1994), [2] M. Medarde, and J. Rodriguez-Carvajal, Z. Phys. B 102, 307 (1997)

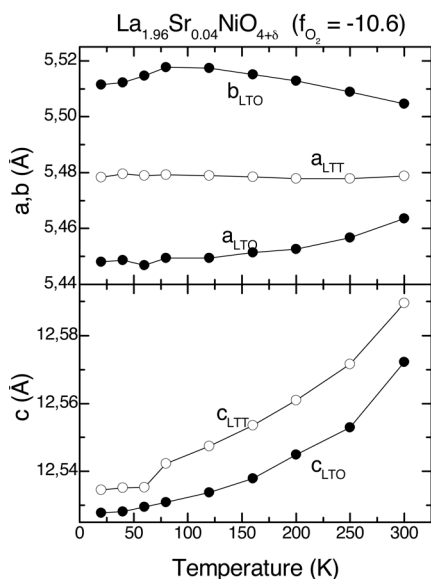


Fig. 1: Lattice parameters  $a, b, c$  of  $\text{La}_{1.96}\text{Sr}_{0.04}\text{NiO}_{4+\delta}$  ( $f_{\text{O}_2} = -10.6$ ) as a function of T.

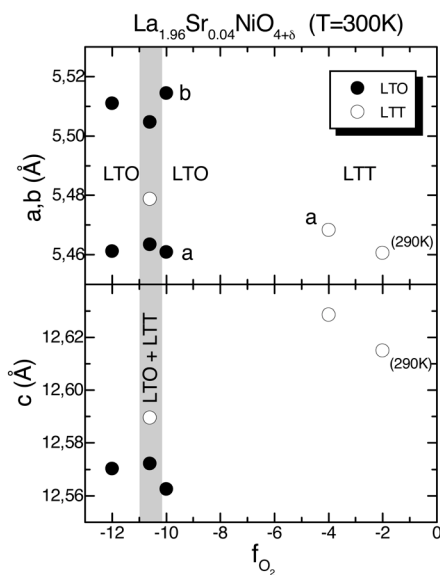


Fig. 2: Lattice parameters  $a, b, c$  of  $\text{La}_{1.96}\text{Sr}_{0.04}\text{NiO}_{4+\delta}$  as a function of the fugacity  $f_{\text{O}_2}$ .